DEVELOPMENT OF NANOCRYSTALLINE $Fe_{80}Cr_{20}$ ALLOY USING COMBINATION TECHNIQUE OF BALL MILLING AND ULTRASONIC TREATMENT FOR FUEL CELL INTERCONNECTOR

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DEDICATION

This thesis is dedicated to my father Mr. Jalis, my mother Ms. Nasi'ah and my sister Ms. Dwi Lusiana that has given their love, enthusiasm and really gave me an energy as well as encouragement when I need it.

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ABSTRACT

Solid Oxide Fuel Cell (SOFC) system consists of anode, cathode, electrolyte and interconnect. This research is focused on interconnect material. The objective of this study is to explore the high energy ball milling (milled) combined with ultrasonic treatment (UT) in obtaining smaller crystallite size, finer surface morphology, higher thermal stability and more homogenous nanocrystalline $Fe_{80}Cr_{20}$ alloys. This condition was motivated by the previous research that some of the grain growth was observed in a high temperature. At first, this process was carried out by high energy ball milling with milling time of 60 h and later, the samples experienced the ultrasonic treatment with frequency of 35 kHz at various periods of 3 h, 3.5 h, 4 h, 4.5 h, and 5 h. Moreover, it was found that there are no works on these combination treatments (milled and UT). Characterization and analysis were carried out to all samples by using X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Energy dispersive X-ray Diffraction (EDS), Thermo Gravimetric Analysis (TGA) and Particle Size Analyzer (PSA). The results showed that the combination treatment samples increases effectively to the solid solubility of Cr to Fe up to 62.1% and decreased the crystallite size up to 2.71 nm at milled and UT 4.5 h sample, these resulted and produces finer surface structure. From EDS results, the combination treatment samples are at suitable composition of 20.05 wt% Cr and 79.95 wt% Fe as compared to other samples. Higher thermal stability was observed on combination treatment sample at 1100 ^{0}C up to 12.7 mg or convenient to 63 wt%, 62 wt% and 25 wt% as compared to raw material, UT samples and milled 60 h sample, respectively. The particle size decreased up to 5.23 µm and particle size distribution of combination treatment relatively increased up to 89.57%. It can be concluded that the combination treatment at milled and UT 4.5 h is appropriate to achieve high solid solubility, nano crystallite size, fine surface morphology, high thermal stability and homogenous $Fe_{80}Cr_{20}$ alloys.

ABSTRAK

Sistem sel fuel oksida pepejal (SOFC) terdiri daripada anod, katod, elektrolit dan plat penyambung. Perkara utama yang ditekankan dalam kajian ini ialah bahan plat penyambung yang digunakan. Objektif kajian ini adalah untuk memperoleh saiz hablur $Fe_{80}Cr_{20}$ yang lebih kecil, permukaan morfologi yang lebih halus, kestabilan terma yang lebih tinggi serta mempunyai nano hablur yang lebih seragam dengan menggunakan mesin bebola pengisar yang digabungkan dengan rawatan ultrasonik. Kaedah ini berdasarkan kajian lepas yang menunjukkan pertumbuhan butiran berlaku pada suhu tinggi. Pada mulanya, proses dimulakan dengan mengisar bahan dengan meggunakan bebola pengisar bertenaga tinggi dalam tempoh 60 jam. Kemudian bahan diberi rawatan ultrasonik dengan frekuensi 35 kHz pada tempoh yang berbeza iaitu 3, 3.5, 4, 4.5 dan 5 jam. Tambahan lagi, sebelum ini tiada kajian yang menggunakan teknik gabungan antara pengisaran dan rawatan ultrasonik. Perincian dan analisis bahan diperolehi menggunakan belauan sinar X (XRD), mikroskop imbasan elektron (SEM), tenaga serakan belauan sinar X (EDS), analisis gravimetrik terma (TGA) dan penganalisis saiz zarah (PSA). Keputusan menunjukkan kaedah penggabungan teknik pengisar dan UT 4.5 jam meningkatkan kelarutan pepejal Cr ke Fe sehingga 62.1 %, mengurangkan saiz hablur sehingga 2.71 nm dan menghasilkan struktur permukaan yang lebih halus. Keputusan daripada EDS, penggabungan rawatan bahan adalah sesuai pada komposisi 20.05 wt% Cr and 79.95 wt% Fe berbanding dengan sampel lain. Penggabungan rawatan bahan pada suhu 1100 ^{0}C mempunyai penambahan berat 12.7 mg yang menunjukkan ketahanan panas yang tinggi berbanding dengan bahan mentah, UT dan pengisar 60 jam menunjukkan pengurangan berat sebanyak 63 wt%, 62 wt% and 25 wt%. Penggabungan rawatan mengurangkan saiz partikel sehingga 5.23 µm dan pengagihan saiz meningkat secara relatif sehingga 89.57%. Secara kesimpulannya penggabungan rawatan pengisar and UT 4.5 jam adalah yang paling sesuai untuk mendapatkan kelarutan pepejal yang tinggi, saiz nano hablur, permukaan morfologi yang halus, kestabilan terma yang tinggi serta mempunyai nano hablur yang seragam.

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LIST OF SYMBOLSAND ABBREVIATION

SOFC	-	Solid Oxide Fuel Cell
SO_x	-	Sulfur Oxide
NOx	-	Nitrogen Oxide
CO_2	-	Carbon Dioxide
CO	-	Carbon Monoxide
CH_4	-	Methane
rad	-	radian
H_2	-	Hydogen
µm	-	Micrometer (10^{-6} meter)
nm	-	Nanometer (10^{-9} meter)
h	-	Hours
YSZ	-	Yttrium Stabilized Zirconia
$LaMnO_3$	-	Lanthanum Manganite
Ni	-	Nickel
H_2O	-	Air
Fe	-	Iron
FeO	-	Iron Oxide
Cr	-	Chromium
Fe-Cr	-	Iron Chromium
Cr_2O_3	-	Chromium Oxide
BCC	-	Body Centered Cubic
FCC	-	Face Centered Cubic
HCP	-	Hexagonal Close-Packed
Å	-	Angstrom
D	-	The diameter crystallite size (nm).
k	-	The shape factor (k=0.9).
λ	-	Wavelength of the X-Ray (λ=0.154056 nm for Cu-K α).

θ	-	Bragg diffraction angle.
B	-	The broadening of the diffraction peaks measured at half of its maximum intensity (in radians).
$B_{corrected}$	-	The instrumental corrected Full Width at Half Maximum (FWHM) in radians
B_{obs}	-	The Full Width at Half Maximum (FWHM) from diffraction peak of the specimens
$B_{instrument}$	-	Standard peaks of the standard reference materials
ε	-	The strain present in the material
d	-	Interplanar spacing (Å)
a	-	Lattice parameter
Xss	-	Solid Solubility (%)
WD	-	Work Distance (mm)
kV	-	Kilo Volt
mm	-	Millimeter
kHz	-	Kilo Hertz
SPS800	-	Spark Plasma Sintering at 800 ^{0}C
UTHM	-	University Tun Hussein Onn Malaysia
TGA	-	Thermo Gravimetric Analysis
EDS	-	Energy Dispersive X-Ray Diffraction
PSA	-	Particle Size Analyzer
FeCrAl	-	Iron Chromium Aluminum
$Fe_{80}Cr_{20}$	-	Composition of alloy powder with 80 wt% Fe and 20 wt% Cr
UT	-	Ultrasonic Treatment
Milled	-	Ball milling technique
Milled and UT	-	Combination technique (Ball milling 60 h and ultrasonic treatment)
MA	-	Mechanical Alloying
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscopy
PCA	-	Process Control Agent

CTE	-	Coefficient of Thermal Expansion
$LaCrO_3$	-	Lanthanum Chromites
LPG	-	Liquefied Petroleum gas
INCO	-	International Nickel Company
HVOF	-	High Velocity Oxy-Fuel
YSZ	-	YttriaStabilizedZirconia
SCZ	-	Scandia Stabilized Zirconia
LSGM	-	Lanthanum Strontium Gallium Magnesium
CGO	-	Cerium Gadolinium Oxide
LSM	-	Lanthanum Strontium Manganite
LSCF	-	$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$
Ni-YSZ	-	Nickel-Yttria Stabilized Zirconia
Ni-SDC	-	Nickel-Samaria Doped Ceria
LCR	-	Lanthanum Chromites/$LaCrO_3$
RSP	-	Rapid Solidification Processing
CCD	-	Charged Coupled Device
DSP	-	Digital Signal Processor
APU	-	Auxiliary Power Units
S/cm	-	Siemens per centimetre

CHAPTER 1

INTRODUCTION

This chapter consists of the research background, problem statement, hypothesis, research objectives and scope of study.

1.1 Research Background

Solid Oxide Fuel Cells (SOFC) research develop most interest currentlyas its potential to create an efficient system, high energy-density power generation device which typically operates at high temperatures relatively (≅1000 ^{0}C). Nowadays, research in SOFC focuses on the interconnect material because it must be stable chemically in both of anode and cathode at temperature approximately 1000^{0}C (Zhang, 2006 and Zhong *et al.*, 2004), as well as grain growth on that temperature was observed by Saryanto *et al.*, (2009); Pudji *et al.*,(2009); Darwin *et al.*,(2010a) and Deni (2011). The interconnect is required to connect thermally the different cells and also providesthe physical barriers to keep the oxidant and fuel separated (Yaodong *et al.*, 2007). Metallic interconnect has interest material to promote as interconnect because their high thermal conductivities (Deni, 2011 and Khaerudini *et al.*, 2012). In the current research, there is high interest in nanocrystalline iron and chromium based alloys. The iron-chromium system has long been used by many engineering alloys as the basis in high-strength and corrosion-resistant applications such as for fuel cell interconnect. Traditionally, iron-based alloy with additions of chromium has been used in high temperature (up to 1000^{0}C) applications (Basu, 2007), where the stabilization and oxidation behavior at high temperature are fundamental in actual applications. Chromium has a phase stabilizer when added into the iron. It is because the chromium has the same BCC crystal structure with iron and made primarily to promote the formation of a dense, adherent layer of Cr_2O_3 on the surface of the alloy (Craig, 1995).

Developing $Fe_{80}Cr_{20}$ based alloy using mechanical alloying via high energy ball milling was successfully carried out by Saryanto *et al.*, (2009). Mechanical alloying (MA) are in general micro and nano structured often have remarkable properties. The product depends on many parameters such as the milling conditions, amount material which are being processed, type of the ball mill used and milling time (Suryanarayana, 2001 and Suryanarayana, 2004). Mechanical alloying technique is able to synthesize single-phase FeT (T = Cr, Cu and Ni) binary alloys with average crystalline size below 50 nm and exhibiting a variety of magnetic responses (Martinez-Blanco *et al.*, 2011). Therefore, it is very challenging to develop nanocrystalline FeCr alloy, because the Cr metal encourage the formation of protective oxide (scale) (Quadakkers *et al.*, 2003). Nanoscale particle research has become a very important field in materials science recently. A nanocrystalline material was characterized by a microstructural length scale in 1 to 100 nm range (Koch, 2003). Nano powders or nano materials with physical, chemical, and the mechanical properties can be utilized as the main building of innovative solutions for the problems in energy, environment, health and communication (Ozlem *et al.*, 2012). Nanoscale particles usually have different physical properties compared with large particles. It had been found that nanoparticles exhibit variety of previously unavailable properties including magnetic, optical, and other physical properties as well as the surface reactivity depending on particle size (Poole *et al.*, 2003). Therefore, heat-resistant nanoscale particle alloys is produced in the industrial scale and widely used in different fields of science and technology.

In the area of ultrasonic processing in liquid media, the development of the power generators family with extensive radiators has strongly contributed to the implementation at semi-industrial and industrial stage of several commercial applications. Some of the applications are in food industry, environment and process for manufacturing (Juan *et al.*, 2010). The use of high-intensity ultrasonic enhances the reactivity of metals as stoichiometric reagents becamean important synthetic technique for many heterogeneous organic and organometallic reactions, especially for the involving reactive metals. In particular for small matter from several nanometers on couple of microns, ultrasonic is very effective in breaking agglomerates, aggregates and even primaries (Thomas *et al.*, 2006; Pudji *et al.*, 2009;

Krisztian *et al.,* 2010 and Darwin *et al.,* 2010b). The mechanism of the rate enhancements in reactions of metals had been done by monitoring the effect of ultrasonic vibration on the kinetics of chemical reactivity of solids, examining the effects of vibration on the surface morphology and size distributions of powders (Suslick, 1994; Kenneth *et al.,* 1999; Pudji *et al.,* 2009 and Darwin *et al.,* 2010b). Ultrasonic treatment also effectively refines the surface morphology on FeCrAl substrate (Yanuandri, 2011 and Ade, 2012).However, the exploration and improvement novel processes enhance the reducing of finer crystallite size and homogenous distribution crystalline size of iron-chromium is still very challenging.

Many researchers field investigate the nano material using ball milling and ultrasonic treatment in mixing raw material (Suryanarayana, 2001and Quadakkers *et al.,* 2003; Suryanarayana, 2004; Saryanto *et al.,* 2009; Pudji *et al.,* 2009; Darwin *et al.,* 2010a; Ade, 2012 and Ozlem *et al.,* 2012). However, unsatisfactory performance still resulted when applied at high temperature operation such as grain growth, rough surface morphology, high oxidation, and the composition not appropriate with working composition. No studies have yet been done on using combination technique between ball milling and ultrasonic treatment. This study focus on the development of iron-chromium alloy powder promoted using combination technique in order to achieve smaller crystallite size, finer the surface morphology, more homogenous and better thermal stability of $Fe_{80}Cr_{20}$ alloys powder.

1.2 Problem statement

The challenge in this research is how to produce the nano range crystallite size, finer surface morphology, high thermal stability and homogenize powders size. Ball milling process is significant to reduce the crystallite size into nano range size. Nevertheless, processing techniques involves high-temperature conditions at longer processing times cause the growth rate of crystallite size inevitable (Suryanarayana, 2001; Suryanarayana, 2003; Quadakkers *et al.,*2003 and Suryanarayana, 2004). In previous research, grain growth from 5.82 nm to 38.51 nm occurred (Hendi, 2011and Deni, 2011). Meanwhile, Ultrasonic process with fixed frequency develop and achieve homogenous and fine grain structure alloys powder (Suslick, 1988; Kenneth *et al.*,1999; Mikko *et al.*, 2004; Thomas Hielscher, 2005; Darwin *et al.*, 2010b; Ade, 2012 and Pudji *et al.*, 2009). According toYanuandri, (2011) and Ade, (2012), the frequency was conducted in 18.52 kHz and holding time of 10, 20, and 30 minutes which were obtained the rough surface and less homogenous. Nanoscale material have many advantages whencompared with large particle. It was proved by Hendi (2011) and Deni (2011) that the cracks in commercial ferritic steel observed after consolidated process at temperature of 900 ^{0}C. It means that large particle have low thermodynamic stability when consolidated at temperature of 900 ^{0}C and above. Therefore, the combination technique of ball milling and untrasonic treatment is needed to obtain smaller grain size, finer surface morphology and more homogenous crystallite sizes in nano range size.

1.3 Hypothesis

The hypothesis consider in this research are:

1. Ball milling will be effective to reduce the powder size into nanometer range size.
2. Ultrasonic treatment will be improved the homogeneity and finer surface of morphology.
3. Combination technique of ball milling and ultrasonic will be developed new $Fe_{80}Cr_{20}$ alloy powder which have smaller crystallite size, more homogenous, finer surface morphology and higher thermal stability.

1.4 Research objectives

Researchobjectives in this study are:

i. To develop nanostructured $Fe_{80}Cr_{20}$ alloy by using ultrasonic treatment, ball milling and combination technique (milled combined with ultrasonic treatment).
ii. To investigate the influence of different ultrasonic treatment time on particle size and particle distribution.
iii. To investigate the influence of combination technique (milled combined with ultrasonic) on powder size, homogeneity, fine surface morphology and thermal stability.

1.5 Scope of study

The scopes of this research are:

1. Raw material is arranged by 80 wt% Fe, and 20 wt% Cr.
2. Developing the nanocrystalline $Fe_{80}Cr_{20}$ alloy is conducted by using ball milling with milling time of 60 h. According to Hendi (2011), that the milling time of 60 h is the most effective to develop nanocrystalline $Fe_{80}Cr_{20}$ alloy as compared to higher milling time of 80 h.
3. Only nitrogen gas is used in high energy ball milling process
4. Ultrasonic process is introduced at appropriate frequency of 35 kHz and holding times of 3 h, 3.5 h, 4 h, 4.5 h and 5 h.
5. The atmospheric pressure while ultrasonic treatment, characterization and analysis process is assumed 1 atm.
6. The microstructure is observed by using Scanning Electron Microscopy (SEM) and the composition of the individual particle is analyzed by using Energy Dispersive X-Ray Spectroscopy (EDS).
7. Phase analyses is conducted by using X-Ray Diffraction (XRD).

8. Thermal analysis is conducted by using Thermo Gravimetric analysis (TGA) up temperature of 1100 ^{0}C which are carried out to the all samples (treated and untreated samples).
9. Determination of particle size and its distribution of particle size is done by using Particle Size Analyzer (PSA).

CHAPTER 2

LITERATURE REVIEW

This literature related to this research is discussed in this chapter. It consists of the theory about Solid Oxide Fuel Cell (SOFC), interconnect, developing nanocystalline material, homogenizing, and refining the surface morphology.

2.1 Introduction of SOFC

According to Zhang (2006), Solid Oxide Fuel Cell (SOFC) is a promising technology for electricity generation. It converts the chemical energy of the fuel gas to electrical energy directly. Therefore, the electrical efficiencies can be achieved through this system. The system can reach efficiency approximately 80% (efficiency of internal combustion engine is no more 40%) when couple it with heat recovering system for cogeneration (heating applications and electric power). Besides, SOFC can replace several fuels and widely implemented to power supplies from small-scale distribution power supplies to large-scale thermal power generation (Blein, 2005).

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